



PATENT APPLICATION IN THE
UNITED STATES PATENT AND TRADE MARK OFFICE

In re application of Sanderson

Docket No. 1-15240

Application No. 09/744,420

Group Art Unit 1762

Confirmation No. 5624

Filed 03/06/2001

Examiner Eric B Fuller

DECLARATION UNDER 37C.F.R. 1.132

Commissioner for Patents
PO Box 1450
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Sir

I, Kevin David Sanderson hereby declare and state

- 1 I am a citizen of the United Kingdom and my current address is 5 Dewberry Field, Upholland, Wigan, WN8 0BQ, Greater Manchester, United Kingdom.
- 2 I have been awarded the degrees of Bachelor of Science and Doctor of Philosophy from Imperial College of Science Technology and Medicine which is part of the University of London. I have extensive experience in the area of thin film coating on glass including specifically metal oxide coatings deposited on a continuous glass ribbon as part of a float glass production process.
- 3 I have been employed by Pilkington plc since 1995 and am currently employed in the position of Principle Project Manager in the area of on line coating of glass.
- 4 I am the sole inventor of the processes claimed in the above application. I have been asked to comment upon the issues raised in the final rejection of the above application dated 03/11/05. To this end I have read that rejection, the art cited as the basis for that rejection and the amended claims which are the subject of that rejection.
- 5 The claims of the patent application are directed to a process for depositing a coating comprising tungsten oxide on-line during a float glass production process. The process comprises directing a gaseous stream comprising tungsten oxyhalide or tungsten chloride and a source of oxygen on to the

surface of a hot glass ribbon. I understand that these claims will be amended prior to the filing of this declaration to specify that the source of oxygen comprises an ester.

- 6 The float glass production process was developed by Pilkington over forty years ago and is now used to produce the vast majority of the flat glass produced anywhere in the world (an estimate of production in 2004 is that 35 million tonnes of flat glass were produced of which 31 million tonnes were produced using the float process). The float process is described e.g. in British Patent GB 769692. A ribbon of molten glass is allowed to flow over the surface of a bath of molten tin. As is stated in GB 769692 a non oxidising atmosphere is maintained over the bath to prevent oxidation of the surface of the molten tin. A slight plenum is preferably maintained in order to prevent the ingress of air.
- 7 On line coating processes of the type claimed in this application are widely used in the industry to deposit various metal oxide coatings on the glass ribbon. They comprise bringing a vapour stream comprising a volatile precursor of the metal oxide and an oxidant into contact with the glass at a point where the temperature of the glass drives the deposition reaction. Most commonly these processes are carried out in the float bath or in the annealing lehr where the temperature of the glass ranges from 400°C to 800°C. These processes are carried out at the pressure prevailing in the bath or lehr i.e. at or just above atmospheric pressure.
- 8 One important factor in assessing the utility of an on line coating process is the rate at which the coating is deposited. Because the float ribbon is continuous and is moving at speeds of from 10 to 15 metres/minute the coating process needs to have a high deposition rate if it is to be useful. The deposition rate required is proportional to the thickness of the coating which is required. It is possible to use more than one coater to deposit the coating but this is undesirable and in any event there is only room for a limited number of coatiers in the bath.
- 9 A second important factor in assessing the utility of an on line process is the volatility and stability of the precursor of the metal oxide. The precursor must be one which is sufficiently volatile at a temperature which is below that at which it undergoes thermal decomposition. Furthermore the precursor should not pre react with the oxidant before the vapour stream comes into contact with the hot glass ribbon. Pre reaction leads to the formation of powdery deposits on the surface of the glass which is not acceptable.
- 10 The present invention is based upon my discovery that tungsten oxide coatings may be deposited on line using tungsten oxy halide or tungsten chloride as a precursor for a tungsten oxide by directing a gas stream comprising the precursor onto the ribbon at a point where the temperature of the ribbon is in the range 500° to 720°C. These precursors are solids which sublime when heated to form a vapour. Surprisingly I have discovered that they can be volatilised in sufficient concentration to form a useful vapour stream and further that when mixed with an oxidant which comprises an ester they do not

pre react. Also the vapour stream thus formed can be brought into contact with the hot glass ribbon to provide processes which have a high deposition rate and can be used to produce a coating of the desired quality over an extended period. Furthermore they can be used to produce coatings having stoichiometric, non-stoichiometric and doped forms. I believe that the claimed processes represent a significant advance in the art and that they are not obvious in the light of the art cited by the Examiner.

- 11 USP 5286520 Proscia is equivalent to EP 54669 which is discussed in the application in suit. Proscia discloses a CVD process which deposits a coating comprising a fluorinated tungsten oxide upon the surface of a glass ribbon produced as part of the float glass process. Proscia discloses the use of tungsten hexafluoride as the tungsten precursor in his process. Tungsten hexafluoride is a volatile liquid which is easily vaporised and as such is suitable for use in a CVD process. These processes can only produce a fluorinated tungsten oxide and they have the disadvantage that the hydrogen fluoride produced in the reaction may significantly attack the surface of the glass at the high temperatures at which the process is carried out. Proscia does not disclose the use of a tungsten chloride or a tungsten oxyhalide as the tungsten precursor as the Examiner acknowledges on page 2 of the Official Action.
- 12 USP 4687560 Tracey describes a plasma deposition process for the production of a tungsten oxide coating on the surface of a glass substrate. Tracey states that plasma deposition occurs when an electrical discharge in a low pressure mixture of volatile reactants causes the formation of a variety of highly energetic species which chemically interact to form stable deposits. Tracey also states that moderate vapour pressure tungsten compounds capable of plasma oxidation may be utilised as reactants and provides a list of examples which includes tungsten chlorides and tungsten oxyhalides.
- 13 As a skilled man I would regard the teachings of Tracey as being unrelated to the present invention or to the disclosure of Proscia. Tracey vaporises his reactants at low temperature in a low pressure chamber. No reaction occurs until power is applied to generate a plasma. As he states he can use any tungsten precursor which has a boiling point of less than 500°C. There is no possibility of pre reaction. Furthermore the deposition rate is not a limiting factor because he simply maintains the power supply until the desired coating has been deposited. Thus the disclosure of Tracey really amounts to a simple recitation of those tungsten compounds which are relatively low boiling. This is not at all sufficient to suggest that they should be employed in place of the tungsten hexafluoride precursor disclosed by Proscia.
- 14 USP 6268019 Florczak discloses a CVD process for the deposition of a doped titanium oxide coating. His processes use titanium tetrachloride as the titanium precursor and he mentions in passing that his invention includes the use of other metal chlorides such as tin tetrachloride, germanium tetrachloride and vanadium tetrachloride in the processes of his invention. Florczak's invention is his discovery that introducing a fluorine dopant decreases the haze of his titanium dioxide films. He speculates that this haze reduction

would be exhibited in other metal oxides. This speculation is not convincing even in relation to tin, germanium and vanadium and I would not interpret it as reading onto other metals including in particular tungsten.

- 15 Florczak emphasises at column 4 lines 42 to 46 that separate streams of reaction gas (oxygen) and carrier gas with titanium tetrachloride vapour must be employed in order to avoid pre reaction. In practice this would be very difficult to achieve in the context of an on line deposition process. In contrast the claims of my application require the formation of a gaseous stream comprising the tungsten precursor and a source of oxygen which is an ester which stream is then brought into contact with the surface of the glass ribbon. This reflects a much reduced tendency for the precursor and oxidant to pre react. The claimed processes have a significant advantage because the reactants can be pre mixed rather than mixing the reactants at the last possible moment.
- 16 The Examiner's assertion that the claimed processes are obvious over Proscia in view of Tracey and Florczak is not in my opinion justified. As a skilled man I would not regard Tracey as being relevant to an atmospheric pressure CVD process. I would not regard as the combined teachings of Tracey and Florczak as teaching the utility of tungsten oxytetrachloride or tungsten chloride as a precursor in the processes of Proscia. The skilled man seeking an on-line process for the deposition of tungsten oxide would have no incentive to refer to these documents.
- 17 USP 604862 Gallego discloses a coated glass substrate wherein the coating comprises a heat absorbing layer which may comprise tungsten oxide. Gallego states that methods of depositing the heat absorbing layer are described for example in EP 523877 and EP 546669. EP 523877 discloses a plasma assisted CVD process and is irrelevant to the present invention of the same reasons as Tracey. EP 546669 is the Proscia reference discussed above.
- 18 Gallego thus effectively recites the teachings of Proscia. The allegation that the invention is obvious over Gallego in view of Tracey and Florczak is not justified for the reasons given above in relation to the rejection over Proscia in view of Tracey and Florczak.
- 19 USP 5385751 Riaz discloses a CVD process for the deposition of a fluorine doped tungsten oxide coating on the surface of a glass substrate. That substrate may be a glass ribbon produced during a float glass process. Riaz teaches the use of a tungsten alkoxide as the tungsten precursor. Proscia is a co inventor of this Riaz citation and this group has apparently looked for volatile tungsten compounds other than the hexafluoride disclosed in Proscia and proposed the use of tungsten alkoxides in this citation. However from Example 1 of Riaz at column 3 line 63 it can be seen that these alkoxide precursors suffer from a tendency to pre reaction because Riaz arranges for them to be delivered in separate streams to the gas surface. The processes of this invention are differentiated from the disclosure of Riaz for the reasons discussed above in relation to Florczak.

- 20 The Examiner asserts that the claimed invention is obvious over Riaz in view of Tracey and Florczak. This assertion is not justified for the same reasons as set out in relation to the rejection over Proscia in view of Tracey and Florczak. Riaz does not teach the applicants precursor and the skilled man would not look to Tracey and Florczak for alternatives.
- 21 The Examiner comments on earlier submissions by the Applicants and states that in his opinion the skilled man would have a reasonable expectation of success for using the precursor of Tracey in the process of Proscia. Further that he would have the knowledge to use the CVD process of Tracey in place of the CVD process of Proscia. These comments are in my view based on an incomplete understanding of the invention.
- 22 Firstly as explained above in the float glass process a continuous glass ribbon is produced which is moving at speeds of 10 to 15 metres per minute. It is clearly impractical to coat the ribbon whilst it is at an elevated temperature under sub atmospheric pressure because of the difficulties of providing a seal on the moving surface of the ribbon.

Secondly the man skilled in the art of on line coating is searching for a precursor which can be volatilised in sufficient quantity, which is not thermally decomposed, which does not pre react significantly with the oxidant which is used and which then reacts quickly when brought into contact with the hot glass surface. He would regard Tracey as irrelevant to these considerations because Tracey does not address these factors. Tracey uses reactants which can be vapourised under low pressure and at low temperatures, Tracey has no issue with pre reaction because the process is carried out at low temperature and the reaction will be initiated by the plasma and he is not concerned with the deposition rate which he obtains because he can continue the process for as long as necessary.

- 23 The present invention provides an on line atmospheric pressure CVD deposition process which provides a significant advance over the known processes including those of Proscia, Riaz and Florczak. The use of the novel combination of a tungsten chloride or tungsten oxyhalide with an oxidant which comprises an ester provides significant advantages which could not have been predicted on the basis of the secondary references including in particular Tracey.
- 24 I declare further that all statements made of my own knowledge are true and that all statements made on information of belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of the United States Code, and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon

Date.....10th August 2005..... Kevin D Sanderson

Yes - D. S. A -